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MOUTHWASH CONTAINING *ASARUM SIEBOLDII* EXTRACT
AND PREPARATION METHOD THEREOF
[Jog'do'ri'pool Chu'chool'moor'uhl Ham'yoo-ha'nuhn
Goo'gang'cheong'jeong'jeh Mit Guh-uhyi Jeh'jeo-bang'beob]

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ABSTRACT

/1*

The present invention relates to a mouthwash and its preparation method. More specifically, it relates to a mouthwash and its preparation method, wherein by containing *Asarum sieboldii* extract, with its extensive anti-microbial properties, bacteria responsible for gum disease, tooth decay, and bad breath are eliminated and plaque is removed, such that various dental and gum diseases are treated and prevented. The mouthwashes hitherto in use are manufactured to contain ingredients for plaque removal or prevention of gum disease. Being mostly synthetic substances, these ingredients may cause side effects to the body, and though they can prevent dental and gum diseases, they have very minimal or no effect in treating diseases that have already appeared.

To overcome the aforementioned problems of the conventional, the present invention provides a mouthwash that includes *Asarum sieboldii* extract in a regular mouthwash composition, thereby having the effect of eliminating bacteria responsible for gum disease, tooth decay, and bad breath and removing plaque as well as treating and preventing gum disease without side effects even when used over a long period of time.

KEYWORD

Asarum sieboldii

* Number in the margin indicates pagination in the foreign text.

SPECIFICATION

Detailed Description of Invention

Object of Invention

Field of Invention and Prior Art

The present invention relates to a mouthwash and its preparation method. More specifically, it relates to a mouthwash and its preparation method, wherein by containing *Asarum sieboldii* extract, with its /2 extensive anti-microbial properties, bacteria responsible for gum disease, tooth decay, and bad breath are eliminated and plaque is removed, such that various dental and gum diseases are treated and prevented.

Asarum sieboldii is an annual belonging to the family Aristolochiaceae. Because its roots and stems are long and slender and it has a slightly pungent taste, it is also called *saeshin* (xixin = thin and acrid) in Oriental medicine. According to the *Dong'yi'bogam* [a classic 1610 book on Korean medicine], holding in one's mouth or drinking water steeped with *Asarum sieboldii* is an effective folk remedy against a cold accompanied by headache, coughing, chronic bronchitis, toothache, etc.

Although *Asarum sieboldii* has long been used by our people as a folk remedy, as with most past folk remedies that were belittled for being unscientific as we underwent modernization after the Japanese colonial period, the pharmacological efficacy of *Asarum sieboldii*, no longer studied, has been almost lost to our people's memory.

When a large amount of water steeped with *Asarum sieboldii* is taken as a folk remedy, nausea or dizziness occurs, and in serious cases, difficulty in breathing as well. However, because the toxicity of *Asarum*

sieboldii is not that strong, even when a large amount of water steeped with *Asarum sieboldii* is taken, there is almost no risk of death.

As a result of persistent research on *Asarum sieboldii*, the present inventors are now able to provide a mouthwash that, through the appropriate use of the components of *Asarum sieboldii*, can remove plaque or bacteria responsible for gum disease, tooth decay, and bad breath.

Among studies on the components of *Asarum sieboldii*, in the Japanese Journal of Pharmacology (81;129,1961, 87;1529,1967), Nagasawa published of having isolated methyleugenol, safrole, β -pinene, and eucarvone from *Asarum sieboldii*. Also, L. Gracza (CA, 62,11626d, 1965) has reported of having discovered methyleugenol's inhibiting effects on positive bacteria in anti-bacterial tests. However, it is difficult to consider methyleugenol, among the ingredients of *Asarum sieboldii*, as the only factor having the effect of treating and preventing gum disease. We surmise that, through combination with other factors thereamong, *Asarum sieboldii* as a whole has the effect of treating and preventing gum disease and eliminating plaque, bad breath, etc.

Mouthwashes hitherto being used are manufactured with the addition of ingredients that prevent gum disease or of ingredients that remove plaque, etc. Despite their effect of preventing different types of dental and gum diseases, their effect in treating a disease that has already appeared is none or does not go beyond a very minimal level.

Further, the primary ingredients added to conventional mouthwashes are mostly manufactured of chemical substances, and hence when the added ingredients are taken in substantial amounts or over a long period of

time, they would have an effect on the relevant diseases, but also upset the balance of the entire body, and worse they may also give rise to fatal side effects.

Recently, pharmaceutical companies have taken great interest in drugs with herbal ingredients, on account of which, with respect to oral health as well, research is being actively done on using herbal ingredients in toothpastes and mouthwashes.

Technical Task Invention Seeks to Accomplish

To overcome the aforementioned problems of conventional mouthwashes, the present invention aims to provide a mouthwash containing *Asarum sieboldii* extract, whereby the bacteria responsible for gum disease, tooth decay, and bad breath are eliminated and plaque is removed.

Another object of the present invention is to provide a mouthwash which, using an extract of the herbal medicine *Asarum sieboldii*, can treat and prevent different types of dental and gum diseases without side effects even when continually used over a long period of time.

Constitution and Operation of Invention

The present invention's mouthwash containing *Asarum sieboldii* extract was prepared according to the processes below.

First: *Asarum sieboldii* is harvested and dried.

Second: The dried *Asarum sieboldii* is mixed with a solvent or a /3 combination of two or more solvents chosen from among acetone, chloroform, n-hexane, cyclo-pentane, a ketone such as cyclo-butanone, an ester such as ethyl acetate, a low alcohol such as n-butanol, isopropyl alcohol, methanol, ethanol, and water, and then heated.

Third: An extract solution is filtered from the *Asarum sieboldii* mixture that was heated in the preceding process.

Fourth: The solvent in the extract solution that was filtered in the preceding process is evaporated to obtain *Asarum sieboldii* extract.

Fifth: 0.3-1.0 wt% of the *Asarum sieboldii* extract that was obtained in the preceding process is added to a regular mouthwash composition to complete the preparation of a mouthwash containing *Asarum sieboldii* extract.

Each of the above processes shall be explained in detail below.

There are slight differences in the efficacy of *Asarum sieboldii* extract depending on where the plant is harvested. However, the *Asarum sieboldii* that grows in Northeast Asia, such as in China, Japan, Taiwan, and Korea, all have almost the same efficacy. Also, depending on when the plant is harvested, there are differences in the amount of extract that can be obtained. To maximize the amount of extract, it is generally best to harvest the plant in August and September.

For drying the harvested *Asarum sieboldii* in the first process, one must use a shady area without sunshine. If it is dried in an area with direct sunlight, active ingredients contained in the *Asarum sieboldii* break down or evaporate, hence lessening its efficacy or making it difficult to obtain the extract in the desired amount. As for the amount of drying, it is preferred that the *Asarum sieboldii* be dried until the water content on its surface is almost evaporated, with the tips of the leaves slightly shriveled. Although determining the amount of drying is not that important in extracting the extract, the amount of solvent

introduced in the following process is determined by the weight of the dried *Asarum sieboldii*. The more adequate the drying is, the lesser the amount of solvent used, and so it is preferred that the drying amount be suitably determined.

For the solvent introduced in the second process, it is preferred that 1.5-2.5 liters of a solvent, or a combination of two or more solvents, from among acetone, chloroform, n-hexane, cyclo-pentane, a ketone such as cyclo-butanone, an ester such as ethyl acetate, a low alcohol such as n-butanol, isopropyl alcohol, methanol, ethanol, and water be added to every 100 g of dried *Asarum sieboldii*. Also, it is preferred that the mixture be heated for about one to three hours at ambient pressure in a mantle installed with a condenser. Since the heating time may be shortened by lowering the atmospheric pressure to less than ambient, when necessary the atmospheric pressure may be decreased to below 0.1 so as to significantly shorten the heating time. The heating temperature is to be appropriately adjusted by taking into account the boiling point of the solvent being used. In general, the components of *Asarum sieboldii* are sufficiently dissolved when heated for about one to three hours under atmospheric pressure. Nevertheless, depending on the situation, the heating time may be slightly shortened or lengthened.

In the third process, a sludge of *Asarum sieboldii* leaves floats on the upper layer of the mixture heated in the preceding process, while the solvent and an extract corresponding to the essence of *Asarum sieboldii* mix in a liquid state on the lower layer. At this juncture, the sludge of *Asarum sieboldii* leaves on the upper layer is removed, while the liquid

mixture remaining on the lower layer is filtered with a suitable filter in order to obtain an extract solution comprising only the *Asarum sieboldii* extract and solvent.

In the fourth process, the extract solution obtained in the preceding process is evaporated at 40-60°C and about 0.005-0.1 atmospheric pressure to obtain the extract. If the evaporation temperature is above 60°C, chemical change may occur in the extract or the main component of the extract may be sucked out together with air when pressure is reduced. If the temperature is below 40°C, the evaporation may take too much time.

The greater the polarity is of the solvent used, the greater the amount of extract obtained through this process. The amount of *Asarum sieboldii* extract varies somewhat depending on the place and time of the *Asarum sieboldii* harvest. Even when a mixture of several solvents is used to obtain the extract, an amount is obtained that is similar to when each of the solvents is used. For example, two liters water and ethanol at a ratio of 90:10-10:90 were added to 100 g of *Asarum sieboldii* that had been dried in the shade. The yield of the extract obtained by this method was similar to the result of singly using water or ethanol as solvent.

In the fifth process, 0.3-1.0 wt% of the *Asarum sieboldii* extract obtained in the preceding process was mixed into a regular mouthwash composition to complete the mouthwash. Since the types of mouthwash used varied and the ingredients also varied according to the type, there were concerns that in adding the *Asarum sieboldii* extract there would be side effects following mixing with those ingredients; however, no such problems

arose in the results of numerous tests. When the *Asarum sieboldii* /4
extract in the mouthwash was less than 0.3 wt%, its effect was
insignificant; at more than 1.0 wt%, the pungent taste in the mouthwash
was so strong that holding it in the mouth was unpleasant. It is preferred
that the *Asarum sieboldii* extract be contained in the amount of 0.5-0.8
wt%.

Below, the present invention is explained in more detail based on
embodiments.

EMBODIMENT 1

Korean *Asarum sieboldii* was harvested and dried in the shade, then
two liters each of various solvents was added to, and mixed with, every
100 g of the dried *Asarum sieboldii*. The mixture was heated for two hours
under atmospheric pressure in a mantle installed with a condenser. Then,
the sludge on the upper part of the heated mixture was removed, while
the liquid residue below was filtered to obtain an *Asarum sieboldii* extract
solution (i.e., solvent and the *Asarum sieboldii* extract dissolved
therein). The solvent in the extract solution was evaporated at 0.008
atmospheric pressure (5 mm Hg) and 50°C to finally obtain the *Asarum
sieboldii* extract (hereafter merely "extract"). The amount of extract
obtained with each solvent is as in Table 1.

TABLE 1

Type of Solvent	Yield(%)	Type of Solvent	Yield(%)
water	10	acetone	3
ethanol	8	chloroform	4
methanol	8	cyclo-butanone	4
isopropyl alcohol	6	cyclo-pentane	2
n-butanol	6	n-hexane	2
ethyl acetate	7		

Though there were differences in the amount of the extract's component obtained using the various solvents above, analysis of each extract confirmed that there was no great difference in the main component among all the extracts. However, considering that large amounts (i.e., a high yield) of the extract does no harm to the body, it is preferred that water or ethanol be used as the solvent in the extraction.

EMBODIMENT 2

The extracts obtained in Embodiment 1 were tested for their degree of toxicity. The testing method involved isolating methyleugenol from the extract, administering the extract and methyleugenol separately to mice, and measuring their lethal doses. The test results measured the LD₅₀ in mice of the extract's active ingredient as 1.2375-0.778 g/kg, while the LD₅₀ in mice of methyleugenol was 247 mg/kg.

In the mouthwash containing the present invention's extract, the amount of contained extract was very little (generally 0.006-0.020% of the lethal active ingredient above). Moreover, since the dose is not orally ingested but simply held in the mouth and then spit out after gargling, it could be ascertained to be of no harm to the body.

Suppose that 0.6 g of extract (0.6%) was added to 100 g of mouthwash. When the amount of mouthwash used the first time is 2 g, the extract contained therein comes to be 0.012 g. When the maximum value of the lethal active ingredient is used as the basis in the above test, the LD₅₀ comes to be 60 g/50 kg, and the amount of extract contained in the above mouthwash comes to be about 1/5000 of the maximum value of the lethal active ingredient.

EMBODIMENT 3

The extract obtained in Embodiment 1 was combined with several different types of mouthwashes currently being sold in the market and the results were measured in order to find out whether there would be an undesirable chemical reaction with the ingredients of the mouthwash and to determine the efficacy of mouthwash mixed with the extract.

In general, the basic ingredients of mouthwash include enzymes that dissolve various food deposits and plaque in the mouth, humectants that maintain the stability and slight viscosity of the mouthwash, surfactants that generate foam to enhance cleaning and refresh the oral cavity, solvents for dissolving and mixing the composition's ingredients, and preservatives for enabling long-term storage. Secondary ingredients include buffering agents, flavor, fragrance, and color.

However, depending on which chemical substances are actually /5 used to possess these ingredients, there are significant differences among mouthwashes. Thus, below, from among mouthwashes currently being sold in the market, four types were selected that had slight differences in their ingredients. The ingredients contained in each type of mouthwash

are as follows.

TYPE A - ingredients (wt%)

Methylbenzethonium chloride - 0.03
Sodium lauroyl sarcosinate - 0.06
Polyoxyethylene-polyoxypropylene block polymer - 0.05
Sodium fluoride (NaF) - 0.02
Glycerin - 10.00
Ethanol solution - 5.00
Flavor - 0.10
Color - 0.10
Distilled water - 84.64

TYPE B - ingredients (wt%)

Citric acid 0.10
Fructose corn syrup - 27.50
Peppermint oil - 0.08
Eucalyptus oil - 0.01
Clove oil - 0.05
Polyoxyethylene(20) sorbitan monostearate - 0.30
Sodium fluoride (NaF) - 0.02
Alcohol - 10.00
Color - 0.20
Distilled water - 61.74

TYPE C - ingredients (wt%)

Benzyl ammonium chloride - 0.03
Sodium lauroyl sarcosinate - 0.06

Polyoxyethylene-polyoxypropylene block polymer - 0.50

Sodium fluoride (NaF) - 0.02

Glycerin - 10.00

Ethanol solution - 5.00

Flavor - 0.10

Color - 0.10

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Distilled water - 84.19

TYPE D - ingredients (wt%)

Cetylpyridinium chloride - 0.10

Citric acid - 0.10

Peppermint oil - 0.075

Eucalyptus oil - 0.025

Clove oil - 0.05

Polyoxyethylene(20) sorbitan monostearate - 0.30

Sodium fluoride (NaF) - 0.02

Alcohol - 10.00

Sodium saccharin - 0.40

Sorbo - 20.00

Color - 0.10

Distilled water - 68.83

The extract was mixed into each type of mouthwash in the amounts of 0.1 wt%, 0.3 wt%, 0.5 wt%, or 1.0 wt%. Then, for a sensory evaluation of taste, as well as for testing plaque removal, gum disease, and bad breath, with respect to the mouthwashes to which the extract was added,

two groups were composed. One group (X) comprised 25 people having gum disease and bad breath; another group (Y) comprised 25 people with plaque. They were allowed to use the various mouthwashes in which the extract was differently combined to gargle three times a day (for one minute each time) over 30 days, at the end of which period the results were measured.

The sensory evaluation was performed on both X and Y groups. With respect to Embodiment 3, all the mouthwashes containing the extract had a slightly pungent, caustic taste, which intensified in proportion to the amount of extract contained. However, except for when the extract content was 3 wt%, the intensity was not so unpleasant, and the more often the mouthwash was used, the greater the tendency to become familiar and habituated to the taste.

Bad-breath testing was performed on Group X. With respect to all the mouthwash types, there was almost no elimination of bad breath when the extract's content was 0.1 wt%, but the effect did increase the higher the content of the extract. However, when the extract's content was 3 wt%, it was almost impossible to gargle the mouth with it, and so no measurements could be made.

Gum-disease testing was performed on Group X. With respect to all the mouthwash types, no effect of treating gum disease could be found when the extract's content was 0.1 wt%. As with above, when the content of the extract was 3 wt%, the subjects were not able to comply with the test due to the intensity of the taste, and so no measurements could be made. In the rest of the cases, all had an effect on alleviating gum disease, and the higher the content of the extract, the greater the curative effect.

The testing on plaque was performed on Group Y. With respect to all the mouthwash types, no effect on plaque removal could be found when the content of the extract was 0.1 wt%. Also, as with the preceding examples, when the extract's content was 3 wt%, the test could not be carried out. In the case of the remaining mouthwashes in which the content of the extract was 0.3 wt%, 0.5 wt%, and 1.0 wt%, there was an effect of plaque removal.

Effect of Invention

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As seen in the embodiments above, the present invention's mouthwash is regular mouthwash to which an extract of *Asarum sieboldii* is added. Regardless of how the ingredients of the mouthwash were composed and whatever the type of mouthwash, by containing 0.3-1.0 wt% of the extract, there was the effect of eliminating bad breath, preventing and treating gum and dental diseases, as well as removing plaque.

Compared to most mouthwashes sold on the market that contain artificially synthesized substances and only have the effect of preventing gum and dental diseases, the present invention's active ingredient is extracted from a plant and has the unique effect of preventing, as well as treating, dental and gum diseases without side effects.

(57) CLAIMS

Claim 1

A mouthwash having 0.3-1.0 wt% *Asarum sieboldii* extract contained in a regular mouthwash composition.

Claim 2

A method of preparing mouthwash containing *Asarum sieboldii* extract, said method comprising

a process of harvesting and drying *Asarum sieboldii*;

a process of mixing said dried *Asarum sieboldii* with a solvent or a combination of two or more solvents chosen from among acetone, chloroform, n-hexane, cyclo-pentane, a ketone such as cyclo-butanone, an ester such as ethyl acetate, a low alcohol such as n-butanol, isopropyl alcohol, methanol, ethanol, and water, and then heating;

a process of filtering an extract solution from said *Asarum sieboldii* mixture heated in the preceding process;

a process of evaporating said solvent contained in said extract solution from the preceding process in order to obtain *Asarum sieboldii* extract;

and

a process of adding 0.3-1.0 wt% of said *Asarum sieboldii* extract obtained in the preceding process to a regular mouthwash composition in order to complete the preparation of a mouthwash containing *Asarum sieboldii* extract.

Claim 3

A method of preparing mouthwash containing *Asarum sieboldii* extract as in Claim 2 wherein said solvent contained in said extract solution is evaporated at 40-60°C and under 0.005-0.1 atmospheric pressure.